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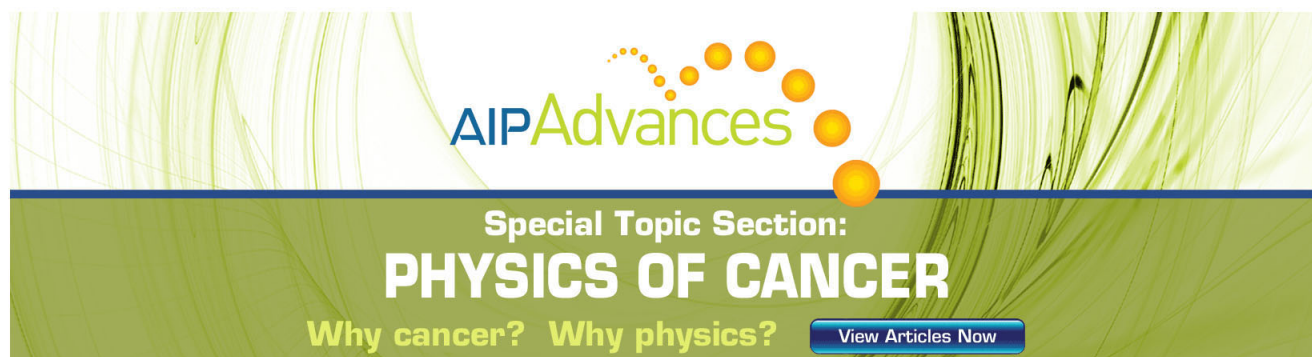
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Enhancement of current-voltage characteristics of multilayer organic light emitting diodes by using nanostructured composite films

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With the aim of improving the photonic efficiency of an organic light emitting diode (OLED) and its display duration, both the hole transport layer (HTL) and the emitting layer (EL) were prepared as nanostructured thin films. For the HTL, nanocomposite films were prepared by spin-coating a homogeneous solution of low molecular weight poly(4-styrenesulfonate) (PEDOT-PSS) and surfactant-capped TiO₂ nanocrystals onto low resistivity indium tin oxide (ITO) substrates; for the EL, nanocrystalline titanium oxide (nc-TiO₂)-embedded Poly[2-methoxy-5-(2'-ethyl-hexyloxy)-1,4-phenylene vinylene] (MEH-PPV+nc-TiO₂) conjugate polymers were spin-coated onto the HTL. Also, for a shallow contact of Al/LiF/MEH-PPV instead of Al/MEH-PPV a super LiF thin film was deposited onto the EL by vacuum evaporation. The resulting multilayer OLED had the following structure of Al/LiF/MEH-PPV+nc-TiO₂/PEDOT-PSS+nc-TiO₂/ITO. Characterization of the nanocomposite films showed that both the current-voltage (*I-V*) characteristics and the photoluminescent properties of the nanocomposite materials were significantly enhanced in comparison with the standard polymers. OLEDs made from these layers would exhibit a large photonic efficiency. © 2009 American Institute of Physics. [DOI: 10.1063/1.3117518]

I. INTRODUCTION

Recently, conducting polymers and polymer-based devices are meeting requirements in many fields such as optoelectronics, organic light emitting diodes (OLEDs), solar flexible cells, etc.¹⁻³ By embedding inorganic nanocrystalline oxides into polymer matrices, one can enhance the efficiency and service duration of the devices. The embedded oxides can substantially influence both the electrical and optical properties of the polymer, for instance, MEH-PPV+nc-TiO₂ composite thin films were studied as a photoactive material.^{4,5} For OLED, it is very important to improve the luminescence efficiency of the emission layer. Efficient device operation requires optimization of three factors: (i) equalization of injection rates of positive (hole) and negative (electron) charge carriers, (ii) recombination of the charge carriers to form singlet exciton in the emitting layer (EL), and (iii) radiative decay of the excitons. Of the two carriers, electrons have the lower mobility and hence limit the current conduction process.⁶ By adding a hole transport layer (HTL) and an electron transport layer (ETL) to the three-layer device one can expect the equalization of injection rates of holes and electrons, consequently leading to a higher electroluminescent efficiency of the OLED. This is because of the better balance of a high working function between the indium tin oxide (ITO) and the HTL, and a low working function between the cathode and the ETL in OLEDs. How-

ever, a large difference in the structures of an inorganic material such as ITO and an organic one such as polyethylene (3,4-dioxythiophene) (PEDOT) usually leads to a poor interface contact. By embedding TiO₂ nanoparticles in PEDOT one can expect to enhance both the contact of HTL with ITO and the working function of PEDOT films. Incorporation of metal (Ni, Cu) and metal-oxide (NiO) nanoparticles in PEDOT:PSS used for both OLEDs and solar cells has also been studied by Oey *et al.*⁷ The results obtained showed that embedding Cu and NiO nanoparticles resulted in inferior OLED performance, while the addition of Ni with 10 and 5 wt % improved the performances for OLEDs and solar cells, respectively. The improvement was attributed to the enhancement of the hole current intensity flowing through the devices. The influence of metallic nanoparticles on the performance of OLEDs has equally been investigated by Choulis *et al.*⁸ Au particles have been incorporated in the interface between alternative buffer layers and a phosphorescence based light emitting layer. It was found that such an incorporation of plasmonic particles led to a 33% improvement in the luminance efficiency of the OLEDs studied. Besides, a super thin LiF layer sandwiched between the EL and the aluminum cathode can create a shallow contact layer (SCL) that enables increasing electron mobility.⁹

In this work we investigated the properties of the nanostructured layer components as proposed resulting in an enhancement observed in the OLEDs. Current-voltage (*I-V*) characteristics of the devices are also presented.

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II. EXPERIMENTAL

The ITO-coated glass substrates used for spin-coating nanocomposite films were ultrasonically cleaned in distilled water, followed by cleaning in ethanol and acetone. KBr crystalline substrates were used for characterization of photoluminescence (PL) properties. To deposit the composite layers onto ITO, MEH-PPV solution was prepared by dissolving MEH-PPV powder in xylene with a ratio of 10 mg of MEH-PPV in 1 ml of xylene. Then, TiO₂ nanoparticles were embedded in these solutions according to a weight ratio TiO₂/MEH-PPV of 0.15 (namely 15 wt %), further referred to as MEHPPV+TiO₂. The last deposit was used as the EL. To obtain a homogeneous dispersion of TiO₂ in polymer, the solutions were mixed for 8 h by using magnetic stirring. These liquid composites were then used for spin-coating and casting. The conditions for spin-coating are as follows: a delay time of 120 s, a rest time of 30 s, a spin speed of 1500 rpm, an acceleration of 500 rpm, and finally a drying time of 2 min. The films used for PL characterization were deposited by casting onto KBr tablets having a diameter of 10 mm, using 50 μ l of the MEH-PPV solution. To dry the films, the samples were put in a flow of dried gaseous nitrogen for 12 h. All the samples were put in a fore vacuum box until the measurements. For preparing an initial solution of PEDOT, the pure PEDOT powder was dissolved in a largely diluted PSS. To get the PEDOT+nc-TiO₂ films that served as the HTL, the above mentioned procedure for MEHPPV+TiO₂ was used, with however a different content of nc-TiO₂ in PEDOT (i.e., 20 wt %). A super thin layer of LiF (thickness of ca. 10 nm) used as a SCL was deposited by electron beam technique, and finally an Al coating used as the cathode was made by vacuum metallization. Thus, the OLED with five layers had the structure of ITO/HTL/EL/SCL/Al.

The surface morphology of samples was characterized by using a "Hitachi" field emission scanning electron microscopy (FE-SEM). Atomic force microscope (AFM) images were obtained using a NT-MDT AFM operating in a tunnel current mode. Nanocrystalline structures were investigated by x-ray diffraction with a Philips D-5005 diffractometer using filtered Cu K α radiation ($\lambda=0.154\ 06$ nm). PL were carried out by using a FL3-2 spectrophotometer and *I-V* characteristics were measured on an Auto-Laboratory Potentiostat PGS-30. The ultraviolet-visible absorption spectra were carried out on a Jasco UV-VIS-NIR V570.

III. RESULTS

A. MEH-PPV+TiO₂ nanocomposites

Surfaces of MEHPPV+TiO₂ nanocomposite samples were examined by SEM and AFM techniques. Figure 1 shows SEM images of a composite sample with embedding of 15 wt % nanocrystalline titanium oxide particles (about 5 nm in size). The surface of this sample appears much smoother than the one of composites with a larger percentage of TiO₂ particles or with larger size TiO₂ particles. The influence of the heat treatment on the morphology of the films was weak, i.e., no noticeable differences in the surface were observed in samples annealed at 120, 150, or 180 $^{\circ}$ C in the same vacuum. However, the most suitable heating tempera-

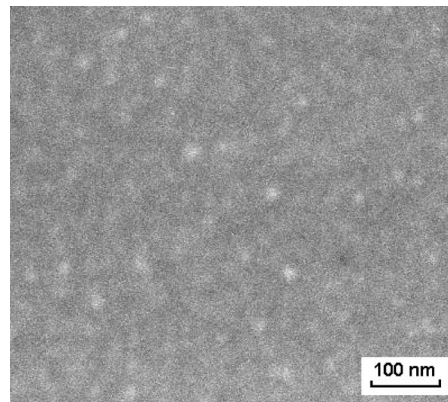


FIG. 1. SEM of MEH-PPV+nc-TiO₂ annealed in vacuum at 150 $^{\circ}$ C.

ture for other properties such as the *I-V* characteristics and the PL spectra was found to be 150 $^{\circ}$ C. In the sample considered, the distribution of TiO₂ nanoparticles is mostly uniform, except for a few bright points indicating the presence of nanoparticle clusters.

The results of PL measurements the MEHPPV+TiO₂ nanocomposite excited at a short wavelength (325 nm) and at a standard one (470 nm) are presented. Figure 2 shows plots of the photoluminescence spectra measured on a pure MEH-PPV and a composite sample, using the FL3-2 spectrophotometer with a He-Ne laser as an excitation source ($\lambda=325$ nm). With such a short wavelength excitation both the polymer and the composite emitted only one broad peak of wavelengths. From this figure, it is seen that the photoemission of the composite film exhibits much higher luminescence intensity than that of the pure MEH-PPV. A blueshift from 580.5 to 550.3 nm was observed for the PL peak, similar to the result in Ref. 10 reporting on the PL spectra for a hybrid MEH-PPV/nc-MoO₃ film. This result is consistent with currently obtained result on polymeric nanocomposites,¹¹ where the blueshift was explained by the reduction of the chain length of polymer, when nanoparticles were embedded in this latter. Although PL enhancement has been rarely mentioned, one can suggest that the increase in the PL intensity for such a composite film can be explained by the large absorption coefficient for TiO₂ particles. Indeed, this phenomenon would be attributed to the nonradiative

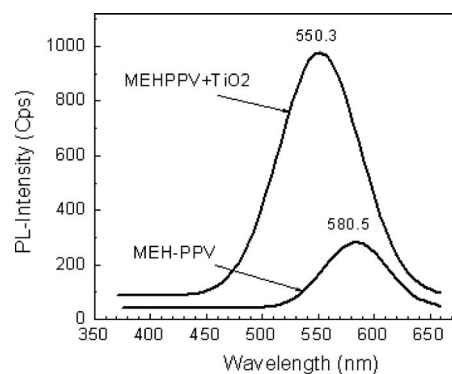


FIG. 2. PL spectra of MEH-PPV+nc-TiO₂. Excitation beam with $\lambda=325$ nm.

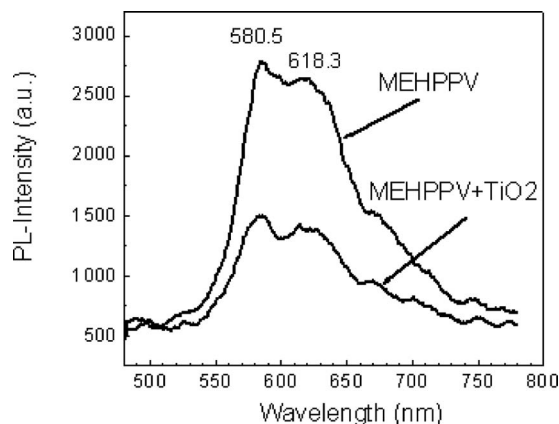


FIG. 3. PL spectra of MEH-PPV+nc-TiO₂. Excitation beam with $\lambda = 470$ nm.

Förster resonant energy transfer¹² from TiO₂ nanoparticles to polymer with excitation of wavelength less than 350 nm.

In Fig. 3 the PL spectra for the MEH-PPV and the composite films with excitation wavelength of 470 nm are plotted. In this case, the MEH-PPV luminescence quenching was observed. For both samples, the photoemission has two broad peaks, respectively, at 580.5 and 618.3 nm. The peak observed at 580.5 nm is larger than the one at 618.3 nm, similar to the EL spectra plotted in Ref. 13. As seen in Ref. 3, for a composite, in the presence of rodlike TiO₂ nanocrystals, PPV quenching of fluorescence is significantly high. This phenomenon was explained by the transfer of the photogenerated electrons to the TiO₂. It is known¹¹ that the fluorescence quenching of MEH-PPV results in charge-separation at interfaces of TiO₂/MEH-PPV, consequently reducing the barrier height at those interfaces.

B. PEDOT+TiO₂ nanocomposites

PEDOT has been used as for the HTL in OLED because it has a high transmission in the visible region, a good thermal stability, and a high conductivity.^{14,15} In the hope to enhance the interface contact between ITO and PEDOT, TiO₂ nanoparticles were embedded into PEDOT in the present work. Figure 4 shows the AFM of a PEDOT composite with a percentage of 20 wt % TiO₂ nanoparticles (about 5 nm in size). With such a high resolution of the AFM

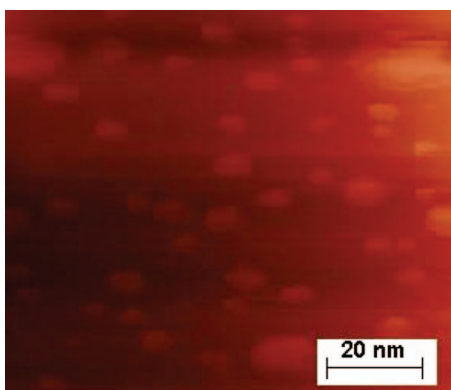


FIG. 4. (Color) AFM of a PEDOT+nc-TiO₂ composite film with embedding of 20 wt % TiO₂ nanoparticles.

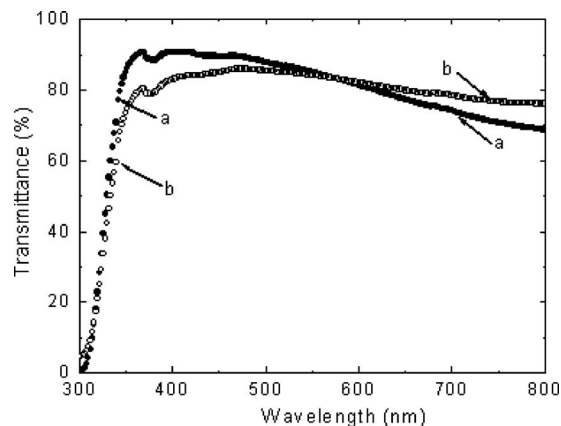


FIG. 5. Transmittance spectra of PEDOT (a) and PEDOT composite films (b).

one can see a distribution of nanoparticles in the polymer due to the spin-coating process. For the pure polymeric PEDOT, the surface exhibits smoothness comparable to the one of the area surrounding the nanoparticles. The TiO₂ nanoparticles contributed to the roughness of the composite surface and created numerous TiO₂/PEDOT boundaries in the composite film.

Transmittance spectra, respectively, for a pure PEDOT and a nanocomposite films are plotted in Fig. 5. From this figure one can see that nanoparticles of TiO₂ made the polymer film more absorbing in the violet range while making it more transparent in the near infrared range. At the range of the emission light of MEH-PPV, namely from 540 to 600 nm, the two samples have about the same transmittance of 82%. This transmittance is a bit lower, but still comparable to the transmittance of the ITO anode. Since PEDOT has a good conductivity, the electrical conductivity of this conducting polymer blend reaching up to 80 S/cm,¹⁶ in the infrared wavelength range it reflects the IR light better resulting in a decrease in the transmittance. The presence of TiO₂ nanoparticles in PEDOT results in a cleavage of the polymer conjugation pathway, consequently leading to a decrease in film conductivity. This is why in the IR range the PEDOT composite has a higher transmittance than that of a pure PEDOT. However, this small decrease in conductivity does not affect much the performance of an OLED that uses the composite as a hole transport layer.

IV. DISCUSSION

To better understand the effect of nanoparticles in composite films used for both the EL and HTL in OLEDs, we measured the *I-V* characteristics of the devices made from different layers, such as a single pure EL diode (ITO/MEH-PPV/Al, abbreviated as SMED), a double pure polymer diode (ITO/PEDOT/MEH-PPV/Al or PPMD), a double polymeric composite layer diode, where a MEH-PPV+TiO₂ composite was used as an EL and a PEDOT-composite film was used as a HTL (ITO/PEDOT+TiO₂/MEH-PPV+TiO₂/Al or PMCD), and a multilayer OLED, where a super thin LiF layer as ETL was added (ITO/PEDOT+TiO₂/MEH-PPV+TiO₂/LiF/Al or MMCD). A 10 nm thick LiF layer used for the SCL was e-beam deposited onto

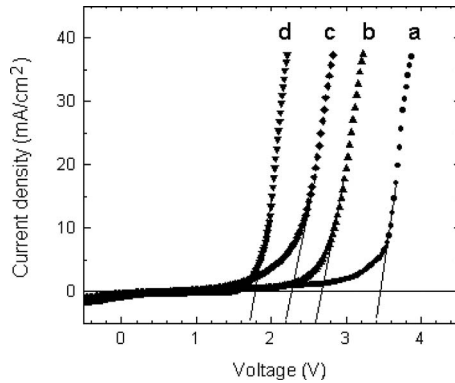


FIG. 6. I - V characteristics of OLED with different laminated structures. (a) Single MEH-PPV, SMED, (b) with HTL layer, PPMD, (c) with HTL and EL composite layers, PMCD, and (d) with LiF, MMCD.

the MEH-PPV+TiO₂; it was then covered by an Al coating prepared by evaporation. A detailed characterization of the SCL was however not carried out here, except for a comparison of the I - V characteristics (Fig. 6). From this figure one can notice the following:

- (i) The turn-on voltages for the diodes SMED, PPMD, PMCD, to MMCD are found to be 3.4, 2.6, 2.2, and 1.7 V, respectively. For the full multilayer diode (MMCD), not only the turn-on voltage but also the reverse current is the smallest. This indicates the equalization of injection rates of holes and electrons due to both the HTL and the SCL, which were added to the OLED.
- (ii) A pure PEDOT used as HTL favors the hole injection from ITO into the organic layer deposited on the HTL, resulting in an enhancement of the I - V characteristics. Thus the turn-on voltage decreased from 3.4 to 2.6 V (see the curve “b” for the PPMD diode).
- (iii) Nanoparticles in both the EL and HTL films have contributed to significantly lowering the turn-on voltage of the device (see the curve “c” for the PMCD diode).

The effect of both the HTL and ETL and/or SCL on the enhancement of the I - V characteristics was well demonstrated, associated with the equalization process of injection rates of holes and electrons. However, the reason why the nanoparticles can improve the device performance is still open for discussion. For instance, in Ref. 17 the authors attributed this enhancement to the stimulated emission of optically pumped MEH-PPV films when TiO₂ particles were embedded in. Whereas in Ref. 5, the authors indicated that no evidence of line narrowing or changes in the line shape was observed at different voltages, implying that the mechanism for improved performance was distinctly different from that found in optically pumped TiO₂/MEH-PPV films. These latter concluded that optical scattering phenomenon was not causing an enhancement in the device performance. Another possible explanation is that the nanoparticle surfaces increase the probability of electron-hole recombination; however, this would result in a change in the external quantum efficiency, rather than the current density as it was observed.

From the data of PL spectra for the MEH-PPV and the

transmittance for PEDOT composites, we have observed both the improvement in PL intensity and the luminescence quenching of the composite (see Figs. 2 and 3). Similar phenomena obtained for nanohybrid layers were explained due to the TiO₂/polymer boundaries causing a difference in band gap between the oxide nanoparticles and the conjugate polymer.¹⁸ Based on these results, we would advance a hypothesis for the improved performance, which supports the suggestion by Carter *et al.*⁵ A change in the device morphology would be caused by the incorporation of nanoparticles into the solution. During the spinning process in the spin-coating technique, the nanoparticles can adhere by strong electrostatic forces to the HTL and between themselves, and capillary forces can then draw the MEH-PPV solution around the nanoparticles into cavities without opening up pinholes through the device. This will result in a rough surface over which the LiF (SCL) is evaporated and subsequently, a large surface area interface between the SCL and the electroluminescent composite material is formed. At a low voltage, charge-injection into MEH-PPV is expected to be cathode limited; the very steep rise in the I - V curves for the composite diodes however, suggests that more efficient injection at the cathode through the SCL is occurring, which would be caused by the rougher interface of the nanocomposites. At a higher voltage, transport in MEH-PPV appears to be space-charge limited.

The electroluminescence quantum efficiency can be calculated by using a simple expression obtained by Tsutsui and Saito in Ref. 19,

$$\eta_{\phi} = \gamma \times \eta_r \times \phi_f, \quad (1)$$

where γ is a double charge injection factor, which is dependent on the processes of carrier injection and is maximal ($\gamma=1$) if a balanced charge injection into the emission layer of the device is achieved, i.e., the number of injected negative charges (electrons) equals the number of injected positive charges (holes); η_r quantifies the efficiency of the formation of a singlet exciton from a positive and a negative polaron, and ϕ_f is the PL quantum efficiency. From the PL spectra and the I - V characteristics obtained, one can see that γ for the MMCD is the largest due to the addition of both the HTL and SCL into the device. Since nc-TiO₂ particles embedded in MEH-PPV constitute a factor favoring electrons faster move in the EL, the intrinsic resistance of the OLED is lowered. This results in an improvement of the I - V characteristics of the device. Moreover, the more mobile electrons can create a larger probability of the electron-hole pairs formation in the emitting layer, resulting in an increase in η_r for the MMCD. Thus the electroluminescence quantum efficiency of the multilayer polymeric composite diodes can be evaluated from Eq. (1) and appears to be much larger than the one for the single polymeric layer device. As a result of the enhanced carriers injection and transport in the polymer composites, the electroluminescence quantum efficiency is roughly estimated to be improved by a factor exceeding 10.

However, to accurately determine the electroluminescence quantum efficiency of a multilayer OLED, we need to carry out more work on the electroluminescence properties of such laminated structures versus the content of nc-TiO₂

particles embedded in both the EL and HTL layers for a larger range of current densities. For completeness, the embedding of nonelectron conducting particles such as SiO₂ will also be investigated. These experiments will be carried out in a setup with parallel measurements of *I-V* and current density-luminescence (*J-L*) characteristics.

V. CONCLUSION

In the hope of enhancing the properties of OLEDs, both the hole transport layer and the emitting layer were prepared as nanostructured thin films by embedding nc-TiO₂ particles into PEDOT and MEH-PPV. The HTL nanocomposite films were spin-coated onto low resistivity ITO substrates. TiO₂-embedded MEH-PPV conjugate polymers were spin-coated onto the HTL. The LiF super thin films serving as SCL were deposited onto the EL by vacuum evaporation. A multilayer OLED of a laminated Al/LiF/MEH-PPV+nc-TiO₂/PEDOT-PSS+nc-TiO₂/ITO structure was prepared for *I-V* measurements. Characterization of the nanocomposite films showed that both the photo- and *I-V* characteristics of the nanocomposite materials were significantly enhanced in comparison with the standard polymers. OLEDs made from these layers may possess a larger electroluminescent efficiency.

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